

PATENT ABSTRACTS OF JAPAN

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(21)Application number : 2000-387638 (71)Applicant : TOKYO OHKA KOGYO CO LTD

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(54) MATERIAL FOR FORMING PROTECTIVE FILM

(57)Abstract:

PROBLEM TO BE SOLVED: To provide a material for forming a protective film in which no void is formed, even if the aspect ratio of a hole is increased and moreover no difference in the film thickness is produced between a Dense part and an Iso part, when the material for forming a protective film is provided thereon.

SOLUTION: In a composition comprising a solid content containing a resin component and a crosslinking agent at a mass ratio of 2:8 to 4:6 and an organic solvent, mass average molecular weight of the solid component is adjusted to be in a range of 1,300-4,500.

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CLAIMS

[Claim(s)]

[Claim 1] The protective coat formation ingredient for dual DAMASHIN processes characterized by adjusting the mass mean molecular weight of the above-mentioned solid content to the range of 1300-4500 in the constituent which consists of solid content which contains a resinous principle and a cross linking agent component at a mass ratio 2:8 thru/or a rate of 4:6, and an organic solvent.

[Claim 2] The protective coat formation ingredient for dual DAMASHIN processes according to claim 1 which has the mass mean molecular weight of the resinous principle in solid content in the range of 4000-15000.

[Claim 3] The protective coat formation ingredient for dual DAMASHIN processes according to claim 1 or 2 whose resinous principle is acrylic resin.

[Claim 4] The protective coat formation ingredient for dual DAMASHIN processes according to claim 1 to 3 whose cross linking agent component is the compound which has triazine structure.

[Claim 5] The protective coat formation ingredient for dual DAMASHIN processes according to claim 4 which has the mass mean molecular weight of a cross linking agent component in the range of 500-1000.

[Translation done.]

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DETAILED DESCRIPTION

[Detailed Description of the Invention]**[0001]**

[Field of the Invention] In the fine structure resist pattern used for a dual DAMASHIN process, in case this invention prepares a protective coat, it relates to the protective coat formation ingredient which can carry out flattening of the thickness difference produced between a high density pattern part and an isolated pattern part.

[0002]

[Description of the Prior Art] The dual DAMASHIN process which embeds a metallic material with plating and forms wiring has come to attract attention instead of processing the conventional metal membrane by etching and forming wiring of an electron device, while using copper as a wiring material is examined, in order to solve the problem of wiring resistance or wiring delay about a semiconductor integrated circuit recently.

[0003] By the way, in preparing a trench hole (wiring gutter) according to this dual DAMASHIN process after forming a beer hall (connection slot), in order for the wiring material already prepared in the substrate front face to prevent being damaged at the time of the trench formation after hole formation, it is necessary to prepare a protective coat in a hole. And about such a protective coat, the pad property for embedding without a clearance the inside of the acid-resisting property of preventing the echo from the substrate of an exposure radiation, and a hole, and the flattening property which makes thickness of the substrate after a pad regularity are demanded.

[0004] By the way, although the organic system ingredient, for example, a photoresist ingredient melttable to an alkali developer, is usually used as a protective coat (JP,10-223755,A) In these organic system ingredients, if the aspect ratio of a hole becomes large Form air bubbles, i.e., a void, in a hole at the time of BEKU, and the protective effect of a metallic material turns to imperfection up, and If it applies on the substrate containing a high density pattern part (henceforth the Dense section), and an isolated pattern part (henceforth the Iso section) and paint film formation is carried out as shown in drawing 1 A thickness difference is produced to a pattern and the fault of having an adverse effect on the lithography processing for consecutive trench formation is produced.

[0005]

[Problem(s) to be Solved by the Invention] This invention conquers the fault which such a conventional protective coat formation ingredient has, and also when it moreover prepares on the Dense section and the Iso section, without forming a void even if the aspect ratio of a hole becomes large, it is made for the purpose of offering the protective coat formation ingredient which does not produce a thickness difference among both.

[0006]

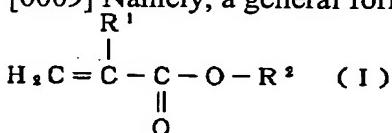
[Means for Solving the Problem] The result of having repeated research wholeheartedly in order that this invention persons might conquer the fault which the conventional protective coat formation ingredient in a dual DAMASHIN process has, If a protective coat is made to form with the ingredient which contained the resinous principle and the cross linking agent component at a specific rate, and the

solid content in it adjusted within the limits of specific mass average molecular weight Formation of the void in a hole can be controlled and it came to make this invention for the ability of thickness between the Dense section and the Iso section to be fixed moreover based on a header and this knowledge. [0007] That is, this invention offers the protective coat formation ingredient for dual DAMASHIN processes characterized by adjusting the mass mean molecular weight of the above-mentioned solid content to the range of 1300-4500 in the constituent which consists of solid content which contains a resinous principle and a cross linking agent component at a mass ratio 2:8 thru/or a rate of 4:6, and an organic solvent.

[0008]

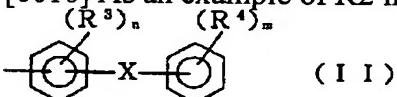
[Embodiment of the Invention] The protective coat formation ingredient of this invention consists of a constituent which dissolved the solid content which consists of a resinous principle and a cross linking agent component in the organic solvent. And the polymer or copolymer obtained especially as the above-mentioned resinous principle, for example, using ester with at least one sort of hydroxy compounds, acrylic acid, or methacrylic acid chosen from the bis-phenyl sulfones which have at least one hydroxyl group, benzophenones, anthracene, and naphthalene as a part of monomer [at least] although a polyamide acid, polysulfone, a halogenation polymer, a polybutene sulfonic acid, acrylic resin, etc. are used is desirable.

[0009] Namely, a general formula [** 1]

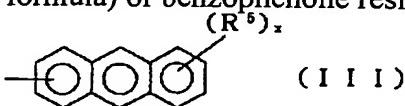


The polymer of the acrylic acid or methacrylic ester expressed with (R1 in a formula is a hydrogen atom or a methyl group, and R2 is the residue excluding the hydrogen atom from the hydroxyl group of the hydroxyl compound chosen from bis-phenyl sulfones with at least one hydroxyl group, benzophenones, anthracene, and naphthalene), these and other acrylic acids, or a copolymer with methacrylic ester is desirable.

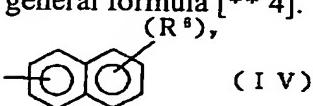
[0010] As an example of R2 in this general formula (I), it is a general formula [** 2].



They are the bis-phenyl sulfone residue expressed with (a -SO₂-radical or a -CO-radical, and R₃ and R₄ are [4 or less and m of a hydrogen atom, hydroxyl-group, alkyl group, alkoxy group, halogen atom, amino-group, low-grade dialkylamino radical, carboxyl group, tert-butoxy radical, tert-butoxycarbonyloxy radical, low-grade alkoxyalkyl group, and low-grade hydroxyalkyl radical, a tetrahydropyranloxy radical or a tetrahydrofuryl oxy-radical, and n] five or less integers for X in a formula) or benzophenone residue, and a general formula [** 3].



They are the anthryl radical expressed with (R5 in a formula is a hydroxyl-group, alkyl group, alkoxy group, halogen atom, amino-group, low-grade dialkylamino radical, carboxyl group, tert-butoxy radical, tert-butoxycarbonyloxy radical, low-grade alkoxyalkyl group, and low-grade hydroxyalkyl radical, a tetrahydropyranyloxy radical, or a tetrahydrofuryl oxy-radical, and x is eight or less integer), or a general formula [** 4].



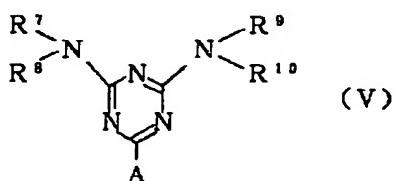
The naphthyl group expressed with (R6 in a formula is a hydroxyl-group, alkyl group, alkoxy group, halogen atom, amino-group, low-grade dialkylamino radical, carboxyl group, tert-butoxy radical, tert-buthoxycarbonyloxy radical, low-grade alkoxyalkyl group, and low-grade hydroxyalkyl radical, a tetrahydropyranloxy radical, or a tetrahydrofuryl oxy-radical, and y is six or less integer) can be mentioned.

[0011] therefore, as an example of the monomer which constitutes a resinous principle A bis(4-hydroxyphenyl) sulfone, a bis(3-hydroxyphenyl) sulfone, A bis(2-hydroxyphenyl) sulfone, a bis(2, 4-dihydroxy phenyl) sulfone, A bis(3, 4-dihydroxy phenyl) sulfone, a bis(3, 5-dihydroxy phenyl) sulfone, A bis(3, 6-dihydroxy phenyl) sulfone, bis(3, 5-dimethyl-4-hydroxyphenyl) sulfones, and these hydroxyl groups leave at least one piece. A tert-butoxy radical, The compound permuted by the tert-buthoxycarbonyloxy radical, the ethoxy ethoxy radical, and the tetra-hydroxy pyranyl oxy-radical, 2, 4-dihydroxy benzophenone, 2 and 3, 4-trihydroxy benzophenone, A 2, 2', 4, and 4'-tetra-hydroxy benzophenone, 2, 2', 5, a 6'-tetra-hydroxy benzophenone, 2-hydroxy-4-methoxybenzophenone, 2-hydroxy-4-octoxybenzophenone, 2-hydroxy-4-dodecyloxy benzophenone, 2, and 2'-dihydroxy-4-methoxybenzophenone, A 2, 6-dihydroxy-4-methoxybenzophenone, 2, 2'-dihydroxy-4, and 4'-dimethoxy benzophenone, A 4-amino-2'-hydroxy benzophenone, a 4-dimethylamino-2'-hydroxy benzophenone, A 4-diethylamino-2'-hydroxy benzophenone, a 4-dimethylamino-4'-methoxy-2'-hydroxy benzophenone, 4-dimethylamino-2' and 4'-dihydroxy benzophenone and 4-dimethylamino-3' and 4'-dihydroxy benzophenones and these hydroxides leave at least one piece. A tert-butoxy radical, The compound permuted by the tert-buthoxycarbonyloxy radical, the ethoxy ethoxy radical, and the tetra-pyranyl oxy-radical, A 1-hydroxy anthracene, a 9-hydroxy anthracene, 1, 2-dihydroxy anthracene, 1, 5-dihydroxy anthracene, 9, 10-dihydroxy anthracene, 1, 2, 3-trihydroxy anthracene, 1, 2 and 3, a 4-tetra-hydroxy anthracene, 1, 2, 3, 4, 5, 6-hexa hydroxy anthracene, 1, 2, 3, 4, 5, 6 and 7, 8-OKUTA hydroxy anthracene, 1-hydroxymethyl anthracene, 9-hydroxymethyl anthracene, 1-hydroxyethyl anthracene, 9-hydroxyethyl anthracene, A 9-hydroxy hexyl anthracene, a 9-hydroxy octyl anthracene, 9, 10-dihydroxy methyl anthracene, 9-anthracene carboxylic acid, A glycidyl-ized anthracene carboxylic acid, glycidyl-ized anthryl methyl alcohol, Anthryl methyl alcohol, a multiple-valued carboxylic acid, for example, oxalic acid, a malonic acid, A methylmalonic acid, an ethyl malonic acid, a dimethyl malonic acid, a succinic acid, a methyl succinic acid, A condensation product with 2 and 2-dimethyl succinic acid, a glutaric acid, an adipic acid, and a pimelic acid, 1-naphthol, 2-naphthol, naphthalene diol, naphthalenetiol, 1-naphthalene methanol, 2-naphthalene methanol, 1-(2-naphthyl) ethanol, a naphthalene carboxylic acid, a 1-naphthol-4-carboxylic acid, 1, 8-naphthalene dicarboxylic acid, naphtholsulfonic acid, etc. can be mentioned. Moreover, as for the substituent expressed with above-mentioned general formula (II) - (IV), it is desirable to use the high thing of absorption to the wavelength of the exposure light used. For example, when i line (365nm) is used, a benzophenone system substituent is desirable, and when a KrF excimer laser (248nm) is used, a sulfone system or an anthracene system substituent is desirable. The mass average molecular weight of these resinous principles has the desirable thing of the range of 4000-15000.

[0012] Moreover, the nitrogen-containing compound which has at least two amino groups permuted by the thing which has the functional group which can form bridge formation between selves or the resinous principle used together with heating as a cross linking agent component, for example, a hydroxyalkyl radical, the alkoxyalkyl group, or its both can be mentioned. As such a compound, there are the melamine and urea with which the hydrogen atom of the amino group was permuted by the methylol radical, the alkoxy methyl group, or its both, for example, guanamine, benzoguanamine, glycoluryl, a succinyl amide, an ethylene urea, etc.

[0013] These nitrogen-containing compounds can be easily obtained making a melamine, a urea, guanamine, benzoguanamine, glycoluryl, a succinyl amide, an ethylene urea, etc. react with formalin in ebullition underwater, and methylol-izing them, or by making lower alcohol still like methyl alcohol, ethyl alcohol, n-propyl alcohol, and isopropyl alcohol react to this, and carrying out alkoxy **.

[0014] In these nitrogen-containing compounds, it is especially a general formula [** 5].



(Although A in a formula shows a hydrogen atom, an alkyl group, an aralkyl radical, an aryl group, or 11R12 -NR(s), it is whether R7, R8, R9, R10, R11, and R12 are mutually the same and a different thing and a hydrogen atom, a methylol radical, or an alkoxy methyl group is shown, respectively) at least two in 4-612 which exist in a molecule, R7, R8, R9, R10, R11, and R12, -- a methylol radical or an alkoxy methyl group -- it is -- since the compound expressed has good crosslinking reaction nature, it is desirable. As for the melamine derivative of the compound expressed with this general formula, what ***** per melamine ring, a methylol radical, or six alkoxy an average of three or more piece methyl groups is desirable. As an example of such a melamine derivative or a benzoguanamine derivative MX-750 by which an average of 3.7 methoxymethyl radicals are permuted per triazine ring of a commercial item, and MW-30 (all are Sanwa chemical company make) by which an average of 5.8 methoxymethyl radicals are permuted per triazine ring, Methoxymethyl-ized melamines, such as Cymel 300, 301, 303, 350, 370, 771, 325, 327, 703, and 712, Methoxymethyl-ized butoxy methylation melamines, such as Cymel 235, 236, 238, 212, 253, and 254, Butoxy methylation melamines, such as Cymel 506 and 508, a carboxyl group content methoxymethyl-ized iso butoxy methylation melamine like Cymel 1141, Methoxymethyl-ized ethoxy methylation benzoguanamine like Cymel 1123, Methoxymethyl-ized butoxy methylation benzoguanamine like Cymel 1123-10, Butoxy methylation benzoguanamine like Cymel 1128, carboxyl group content methoxymethyl-ized ethoxy methylation benzoguanamine (all are the Mitsui Cyanamid make) like Cymel 1125-80, etc. are mentioned. Moreover, as an example of glycoluryl, butoxy methylation glycoluryl like Cymel 1170, methylol-ized glycoluryl like Cymel 1172, etc. are mentioned.

[0015] Moreover, in this invention, these cross linking agents may be used independently, and two or more sorts may be combined and they may be used. As for these cross linking agent components, it is desirable that the mass average molecular weight uses the thing of 500-1000.

[0016] Moreover, 1300-4500, and that it is especially 2000-4000 have the desirable mass average molecular weight of solid content which consists of the above-mentioned resinous principle and a cross linking agent component in this invention. In addition, if this range is exceeded, the clearance called a void will be generated and the function as a protective coat will be reduced to the interior of a hole. Moreover, if less than this range, between Dense and Iso, a thickness difference will arise and control of subsequent etching will become very difficult. The mass mean molecular weight of this solid content is obtained by GPC measurement (polystyrene system gel is used for a column and it is a product made from SHODEX, and "GPC SYSTEM-21" in equipment).

[0017] As an organic solvent in this invention, if the two above-mentioned component may be dissolved, what kind of thing may be used and there will be especially no limit. As an example of such a thing, an acetone, a methyl ethyl ketone, cyclopentanone, Ketones, such as a cyclohexanone, methyl isoamyl ketone, 2-heptanone, 1 and 1, and a 1-trimethyl acetone Ethylene glycol and ethylene glycol mono-acetate, diethylene-glycol, or diethylene-glycol mono-acetate, Or polyhydric alcohol, such as these monomethyl ether, the monopropyl ether, the monobutyl ether, or the monophenyl ether, and the derivative of those, There is ester, such as cyclic ether like dioxane, and ethyl lactate, methyl acetate, ethyl acetate, butyl acetate, methyl pyruvate, pyruvic-acid ethyl, 3-methoxy methyl propionate, 3-ethoxy ethyl propionate. These may be used independently, and may mix and use two or more sorts.

[0018] Moreover, into the protective coat formation ingredient of this invention, a surfactant can also be added by request for improvement in spreading nature, or striae SHON prevention. As such a surfactant, fluorochemical surfactants, such as Sir chlorofluorocarbon SC-103, SR-100 (Asahi Glass Co., Ltd. make), EF-351 (northeast fertilizer company make), Fluorad Fc-431, Fluorad Fc-135, Fluorad Fc-98, Fluorad Fc-430, and Fluorad Fc-176 (Sumitomo 3 M company make), can be mentioned, for example. It is

desirable to choose in less than 2000 ppm to the solid content of the solution for formation of an antireflection film layer as an addition in this case.

[0019] Although the protective coat formation ingredient of this invention is suitable for applying on the substrate which has the pattern which consists of the Dense section and the Iso section As the relation between this Dense section and the Iso section is shown in drawing 2 That in which the Iso section which consists of a hole which carried out ***** isolation of the fixed distance which the hole whose Dense section is plurality crowds in the distance within about 2 to 3 times or it of the diameter of the hole, and is beyond the aforementioned distance is prepared is said.

[0020]

[Example] Next, an example and the example of a comparison explain this invention to a detail further. In addition, three holes with a diameter of 250nm are prepared as a Dense section at spacing which is 500nm, and the sample of the hole pattern of the Dense section-Iso section used in each example separates 5000nm from the hole of the outermost part of the Dense section as an Iso section, and has the same hole as the above.

[0021] The mass average molecular weight whose mass average molecular weight which is acrylic resin which has example 1 dihydroxy phenyl sulfone structure is PAC102 (die toe company make) of 5821 and a melamine derivative dissolved the mixture [of 599] of MX-750 (Sanwa chemical company make) of 6:4 in propylene glycol, made this solid content concentration 2 mass %, and the protective coat formation ingredient was prepared. The solid content mass average molecular weight in this case was 4226. Moreover, spinner spreading of the above-mentioned protective coat formation ingredient was carried out, and the protective coat was made to form on a hole pattern. [to the substrate with which the hole pattern of the Dense section-Iso section as shown in drawing 2 was formed] [180 degrees C] [for 90 seconds] When the sectional view was observed by SEM (scanning electron microscope), there was no generating of the void inside a pattern and the thickness difference between the Dense section-Iso section patterns was about 243nm.

[0022] The protective coat was made to form on a hole pattern by the same actuation as an example 1 except having set to 4:6 PAC102 which used in the example 2 example 1, and the blending ratio of coal of MX-750. The solid content mass average molecular weight in this case was 3235. Consequently, there was also no generating of a void into a pattern and the thickness difference between the Dense section-Iso sections was about 209.5nm.

[0023] The protective coat was made to form on a hole pattern by the same actuation as an example 1 except having set to 8:2 PAC102 which used in the example of comparison 1 example 1, and the blending ratio of coal of MX-750. The solid content mass average molecular weight in this case was 5086. Consequently, since the void occurred in the hole, a hole was not able to be embedded thoroughly.

[0024] The protective coat was made to form on a hole pattern by the same actuation as an example 1 except having set to 1:9 PAC102 which used in the example of comparison 2 example 1, and the blending ratio of coal of MX-750. The solid content mass average molecular weight in this case was 1268. Consequently, although there was no generating of a void into a pattern, the thickness difference between the Dense section-Iso sections was set to 425.2 micrometers, and became the failure of the lithography process after it.

[0025]

[Effect of the Invention] If the protective coat formation ingredient of this invention is used, when preparing a protective coat on the fine structure resist pattern formed in the dual DAMASHIN process, flattening of the thickness difference produced between the Dense section and the Iso section can be carried out, and lithography processing for consecutive trench formation can be performed smoothly.

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DESCRIPTION OF DRAWINGS

[Brief Description of the Drawings]

[Drawing 1] The sectional view showing the condition of the protective coat in the conventional approach.

[Drawing 2] The sectional view of the resist pattern which has the Dense section and the Iso section.

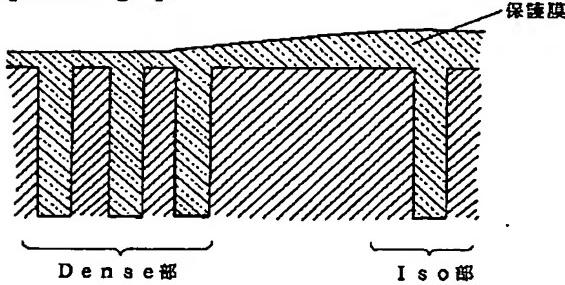
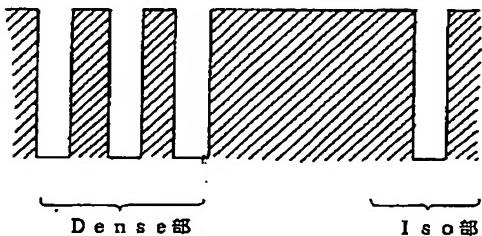
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DRAWINGS

[Drawing 1]**[Drawing 2]**

[Translation done.]

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CORRECTION OR AMENDMENT

[Kind of official gazette] Printing of amendment by the convention of 2 of Article 17 of Patent Law

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H01L 21/768
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[FI]

H01L 21/90 Z
21/312 A

[Procedure amendment]

[Filing Date] January 27, Heisei 15 (2003. 1.27)

[Procedure amendment 1]

[Document to be Amended] Description

[Item(s) to be Amended] 0011

[Method of Amendment] Modification

[Proposed Amendment]

[0011] Therefore, it is an example of the monomer which constitutes a resinous principle, A bis(4-hydroxyphenyl) sulfone, a bis(3-hydroxyphenyl) sulfone, A bis(2-hydroxyphenyl) sulfone, a bis(2, 4-dihydroxy phenyl) sulfone, A bis(3, 4-dihydroxy phenyl) sulfone, a bis(3, 5-dihydroxy phenyl) sulfone, A bis(3, 6-dihydroxy phenyl) sulfone, bis(3, 5-dimethyl-4-hydroxyphenyl) sulfones, and these hydroxyl groups leave at least one piece. A tert-butoxy radical, The compound permuted by the tert-butoxycarbonyloxy radical, the ethoxy ethoxy radical, and the tetrahydropyranloxy radical, 2, 4-dihydroxy benzophenone, 2 and 3, 4-trihydroxy benzophenone, A 2, 2', 4, and 4'-tetra-hydroxy benzophenone, 2, 2', 5, a 6'-tetra-hydroxy benzophenone, 2-hydroxy-4-methoxybenzophenone, 2-hydroxy-4-octoxybenzophenone, 2-hydroxy-4-dodecyloxy benzophenone, 2, and 2'-dihydroxy-4-methoxybenzophenone, A 2, 6-dihydroxy-4-methoxybenzophenone, 2, 2'-dihydroxy-4, and 4'-dimethoxy benzophenone, A 4-amino-2'-hydroxy benzophenone, a 4-dimethylamino-2'-hydroxy benzophenone, A 4-diethylamino-2'-hydroxy benzophenone, a 4-dimethylamino-4'-methoxy-2'-hydroxy benzophenone, 4-dimethylamino-2' and 4'-dihydroxy benzophenone and 4-dimethylamino-3' and 4'-dihydroxy benzophenones and these hydroxyl groups leave at least one piece. A tert-butoxy radical, The

compound permuted by the tert-butoxycarbonyloxy radical, the ethoxy ethoxy radical, and the tetrahydropyranloxy radical, A 1-hydroxy anthracene, a 9-hydroxy anthracene, 1, 2-dihydroxy anthracene, 1, 5-dihydroxy anthracene, 9, 10-dihydroxy anthracene, 1, 2, 3-trihydroxy anthracene, 1, 2 and 3, a 4-tetra-hydroxy anthracene, 1, 2, 3, 4, 5, 6-hexa hydroxy anthracene, 1, 2, 3, 4, 5, 6 and 7, 8-OKUTA hydroxy anthracene, 1-hydroxymethyl anthracene, 9-hydroxymethyl anthracene, 1-hydroxyethyl anthracene, 9-hydroxyethyl anthracene, A 9-hydroxy hexyl anthracene, a 9-hydroxy octyl anthracene, 9, 10-dihydroxy methyl anthracene, 9-anthracene carboxylic acid, A glycidyl-ized anthracene carboxylic acid, glycidyl-ized anthryl methyl alcohol, Anthryl methyl alcohol, a multiple-valued carboxylic acid, for example, oxalic acid, a malonic acid, A methylmalonic acid, an ethyl malonic acid, a dimethyl malonic acid, a succinic acid, a methyl succinic acid, A condensation product with 2 and 2-dimethyl succinic acid, a glutaric acid, an adipic acid, and a pimelic acid, 1-naphthol, 2-naphthol, naphthalene diol, naphthalenetriol, 1-naphthalene methanol, 2-naphthalene methanol, 1-(2-naphthyl) ethanol, a naphthalene carboxylic acid, a 1-naphthol-4-carboxylic acid, 1, 8-naphthalene dicarboxylic acid, naphtholsulfonic acid, etc. can be mentioned. Moreover, as for the substituent expressed with above-mentioned general formula (II) - (IV), it is desirable to use the high thing of absorption to the wavelength of the exposure light used. For example, when i line (365nm) is used, a benzophenone system substituent is desirable, and when a KrF excimer laser (248nm) is used, a sulfone system or an anthracene system substituent is desirable. The mass average molecular weight of these resinous principles has the desirable thing of the range of 4000-15000.

[Procedure amendment 2]

[Document to be Amended] Description

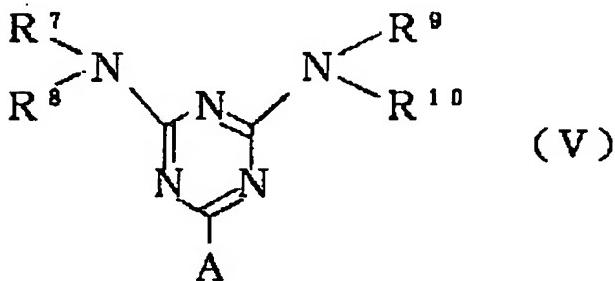
[Item(s) to be Amended] 0014

[Method of Amendment] Modification

[Proposed Amendment]

[0014] In these nitrogen-containing compounds, it is especially a general formula.

[Formula 5]



(A in a formula showing a hydrogen atom, an alkyl group, an aralkyl radical, an aryl group, or 11R12-NR(s), and being whether R7, R8, R9, R10, R11, and R12 are mutually the same and a different thing.) at least two in 4-612 which exist in a molecule although a hydrogen atom, a methylol radical, or an alkoxy methyl group is shown, respectively, R7, R8, R9, R10, R11, and R12, -- a methylol radical or an alkoxy methyl group -- it is -- since the compound expressed has good crosslinking reaction nature, it is desirable. As for the triazine derivative of the compound expressed with this general formula, what ***** per triazine ring, a methylol radical, or six alkoxy an average of three or more piece methyl groups is desirable. As the example of such a triazine derivative or a benzoguanamine derivative, MX-750 by which an average of 3.7 methoxymethyl radicals are permuted per triazine ring of a commercial item, and MW-30 (all are Sanwa chemical company make) by which an average of 5.8 methoxymethyl radicals are permuted per triazine ring, Methoxymethyl-ized melamines, such as Cymel 300, 301, 303, 350, 370, 771, 325, 327, 703, and 712, Methoxymethyl-ized butoxy methylation melamines, such as Cymel 235, 236, 238, 212, 253, and 254, Butoxy methylation melamines, such as Cymel 506 and 508, a carboxyl group content methoxymethyl-ized iso butoxy methylation melamine like Cymel 1141, Methoxymethyl-ized ethoxy methylation benzoguanamine like Cymel 1123, Methoxymethyl-ized

butoxy methylation benzoguanamine like Cymel 1123-10, Butoxy methylation benzoguanamine like Cymel 1128, carboxyl group content methoxymethyl-ized ethoxy methylation benzoguanamine (all are the Mitsui Cyanamid make) like Cymel 1125-80, etc. are mentioned. Moreover, as an example of glycoluryl, butoxy methylation glycoluryl like Cymel 1170, methylol-ized glycoluryl like Cymel 1172, etc. are mentioned.

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最終頁に続く

(54)【発明の名称】 保護膜形成材料

(57)【要約】

【課題】 ホールのアスペクト比が大きくなつてもボイドを形成することなく、しかもDense部とIso部の上に設けた場合にも、両者間に膜厚差を生じることのない保護膜形成材料を提供する。

【解決手段】 樹脂成分と架橋剤成分とを質量比2:8ないし4:6の割合で含む固形分と有機溶剤からなる組成物において、上記固形分の質量平均分子量を1300～4500の範囲に調整する。

1

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【特許請求の範囲】

【請求項1】樹脂成分と架橋剤成分とを質量比2:8ないし4:6の割合で含む固形分と有機溶剤からなる組成物において、上記固形分の質量平均分子量を1300～4500の範囲に調整したことを特徴とするデュアルダマシンプロセス用保護膜形成材料。

【請求項2】固形分中の樹脂成分の質量平均分子量が4000～15000の範囲にある請求項1記載のデュアルダマシンプロセス用保護膜形成材料。

【請求項3】樹脂成分がアクリル系樹脂である請求項1又は2記載のデュアルダマシンプロセス用保護膜形成材料。

【請求項4】架橋剤成分がトリアジン構造を有する化合物である請求項1ないし3のいずれかに記載のデュアルダマシンプロセス用保護膜形成材料。

【請求項5】架橋剤成分の質量平均分子量が500～1000の範囲にある請求項4記載のデュアルダマシンプロセス用保護膜形成材料。

【発明の詳細な説明】

【0001】

【発明の属する技術分野】本発明は、デュアルダマシンプロセスに用いる微細構造レジストパターンにおいて、保護膜を設ける際に、密集パターン部分と孤立パターン部分との間に生じる膜厚差を平坦化しうる保護膜形成材料に関するものである。

【0002】

【従来の技術】最近、半導体集積回路に関し、配線抵抗又は配線遅延の問題を解決するために、配線材料として銅を用いることが検討されるとともに、従来の金属膜をエッチングにより加工して電子デバイスの配線を形成する代わりに、金属材料をめっきにより埋め込んで配線を形成するデュアルダマシンプロセスが注目されるようになってきた。

【0003】ところで、このデュアルダマシンプロセスにより、ピアホール（接続溝）を形成した後で、トレンチホール（配線溝）を設ける場合には、基板表面に既に設けられている配線材料がホール形成後のトレンチ形成時に損傷するのを防ぐために、ホール内に保護膜を設ける必要がある。そして、このような保護膜については、露光放射線の基板からの反射を防止する反射防止特性、ホール内を隙間なく埋め込むための埋込み特性、及び埋込み後の基板の膜厚を一定にする平坦化特性が要求されている。

【0004】ところで、通常、保護膜としては、有機系材料、例えばアルカリ現像液に可溶なホトレジスト材料が用いられているが（特開平10-223755号公報）、これらの有機系材料においては、ホールのアスペクト比が大きくなると、ベーク時に気泡、すなわちボイドをホール内に形成し、金属材料の保護効果が不十分になる上に、図1に示すように密集パターン部分（以下D

ense部という）と孤立パターン部分（以下iso部という）とを含む基板上に塗布して塗膜形成させると、パターンに膜厚差を生じ、後続のトレンチ形成のためのリソグラフィー処理に悪影響を与えるという欠点を生じる。

【0005】

【発明が解決しようとする課題】本発明は、このような従来の保護膜形成材料のもつ欠点を克服し、ホールのアスペクト比が大きくなてもボイドを形成することなく、しかもDense部とiso部の上に設けた場合にも、両者間に膜厚差を生じることのない保護膜形成材料を提供することを目的としてなされたものである。

【0006】

【課題を解決するための手段】本発明者らは、デュアルダマシンプロセスにおける従来の保護膜形成材料のもつ欠点を克服するために鋭意研究を重ねた結果、樹脂成分と架橋剤成分とを特定の割合で含み、かつその中の固形分が特定の質量平均分子量の範囲内に調整した材料で保護膜を形成せれば、ホール内におけるボイドの形成を抑制することができ、しかもDense部とiso部との間の膜厚を一定にしうることを見出し、この知見に基づいて本発明をなすに至った。

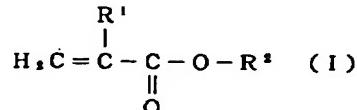
【0007】すなわち、本発明は、樹脂成分と架橋剤成分とを質量比2:8ないし4:6の割合で含む固形分と有機溶剤からなる組成物において、上記固形分の質量平均分子量を1300～4500の範囲に調整したことを特徴とするデュアルダマシンプロセス用保護膜形成材料を提供するものである。

【0008】

【発明の実施の形態】本発明の保護膜形成材料は、樹脂成分と架橋剤成分とからなる固形分を有機溶剤に溶解した組成物からなっている。そして、上記の樹脂成分としては、例えば、ポリアミド酸、ポリスルホン、ハロゲン化重合体、ポリブテンスルホン酸、アクリル系樹脂などが用いられるが、中でも、少なくとも1個の水酸基を有するビスフェニルスルホン類、ベンゾフェノン類、アントラセン類及びナフタレン類の中から選ばれた少なくとも1種のヒドロキシ化合物とアクリル酸又はメタクリル酸とのエステルをモノマーの少なくとも一部として用いて得られる重合体又は共重合体が好ましい。

【0009】すなわち、一般式

【化1】

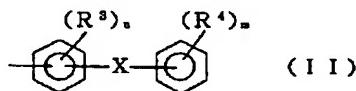


（式中のR'は水素原子又はメチル基であり、R''は少なくとも1個の水酸基をもつビスフェニルスルホン類、ベンゾフェノン類、アントラセン類及びナフタレン類の中から選ばれたヒドロキシ化合物の水酸基から水素原子

を除いた残基である)で表わされるアクリル酸又はメタクリル酸エステルの重合体あるいはこれらと他のアクリル酸又はメタクリル酸エステルとの共重合体が好ましい。

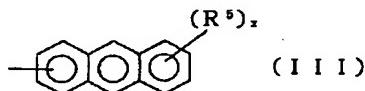
【0010】この一般式(I)中のR²の例としては、一般式

【化2】



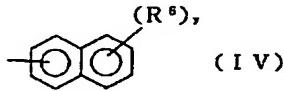
(式中のXは-SO₂-基又は-CO-基、R³及びR⁴は水素原子、水酸基、アルキル基、アルコキシ基、ハロゲン原子、アミノ基、低級ジアルキルアミノ基、カルボキシル基、tert-ブトキシ基、tert-ブトキカルボニルオキシ基、低級アルコキシアルキル基、低級ヒドロキシアルキル基、テトラヒドロピラニルオキシ基又はテトラヒドロフラニルオキシ基、nは4以下、mは5以下の整数である)で表わされるビスフェニルスルホン残基又はベンゾフェノン残基、一般式

【化3】



(式中のR⁵は水酸基、アルキル基、アルコキシ基、ハロゲン原子、アミノ基、低級ジアルキルアミノ基、カルボキシル基、tert-ブトキシ基、tert-ブトキカルボニルオキシ基、低級アルコキシアルキル基、低級ヒドロキシアルキル基、テトラヒドロピラニルオキシ基又はテトラヒドロフラニルオキシ基、xは8以下の整数である)で表わされるアントリル基、あるいは一般式

【化4】



(式中のR⁶は水酸基、アルキル基、アルコキシ基、ハロゲン原子、アミノ基、低級ジアルキルアミノ基、カルボキシル基、tert-ブトキシ基、tert-ブトキカルボニルオキシ基、低級アルコキシアルキル基、低級ヒドロキシアルキル基、テトラヒドロピラニルオキシ基又はテトラヒドロフラニルオキシ基、yは6以下の整数である)で表わされるナフチル基を挙げができる。

【0011】したがって、樹脂成分を構成するモノマーの例としては、ビス(4-ヒドロキシフェニル)スルホン、ビス(3-ヒドロキシフェニル)スルホン、ビス(2-ヒドロキシフェニル)スルホン、ビス(2,4-ジヒドロキシフェニル)スルホン、ビス(3,4-ジヒ

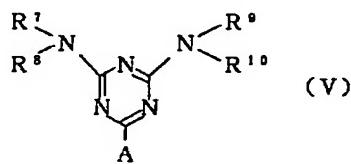
ドロキシフェニル)スルホン、ビス(3,5-ジヒドロキシフェニル)スルホン、ビス(3,6-ジヒドロキシフェニル)スルホン及びこれらの水酸基が少なくとも1個を残してtert-ブトキシ基、tert-ブトキカルボニルオキシ基、エトキシエトキシ基、テトラヒドロキシピラニルオキシ基で置換された化合物、2,4-ジヒドロキシベンゾフェノン、2,3,4-トリヒドロキシベンゾフェノン、2,2',4,4'-テトラヒドロキシベンゾフェノン、2,2',5,6'-テトラヒドロキシベンゾフェノン、2-ヒドロキシ-4-メトキシベンゾフェノン、2-ヒドロキシ-4-オクトキシベンゾフェノン、2-ヒドロキシ-4-デシルオキシベンゾフェノン、2,2'-ジヒドロキシ-4-メトキシベンゾフェノン、2,6-ジヒドロキシ-4-メトキシベンゾフェノン、2,2'-ジヒドロキシ-4,4'-ジメトキシベンゾフェノン、4-アミノ-2'-ヒドロキシベンゾフェノン、4-ジメチルアミノ-2'-ヒドロキシベンゾフェノン、4-ジエチルアミノ-2'-ヒドロキシベンゾフェノン、4-ジメチルアミノ-4'-メトキシ-2'-ヒドロキシベンゾフェノン、4-ジメチルアミノ-2',4'-ジヒドロキシベンゾフェノン、4-ジメチルアミノ-3',4'-ジヒドロキシベンゾフェノン及びこれらの水酸化物が少なくとも1個を残してtert-ブトキシ基、tert-ブトキカルボニルオキシ基、エトキシエトキシ基、及びテトラピラニルオキシ基で置換された化合物、1-ヒドロキシアントラセン、9-ヒドロキシアントラセン、1,2-ジヒドロキシアントラセン、1,5-ジヒドロキシアントラセン、9,10-ジヒドロキシアントラセン、1,2,3-トリヒドロキシアントラセン、1,2,3,4-テトラヒドロキシアントラセン、1,2,3,4,5,6-ヘキサヒドロキシアントラセン、1,2,3,4,5,6,7,8-オクタヒドロキシアントラセン、1-ヒドロキシメチルアントラセン、9-ヒドロキシメチルアントラセン、1-ヒドロキシエチルアントラセン、9-ヒドロキシエチルアントラセン、9-ヒドロキシヘキシルアントラセン、9-ヒドロキシオクチルアントラセン、9,10-ジヒドロキシメチルアントラセン、9-アントラセンカルボン酸、グリシジル化アントラセンカルボン酸、グリシジル化アントリルメチルアルコール、アントリルメチルアルコールと多価カルボン酸、例えばシュウ酸、マロン酸、メチルマロン酸、エチルマロン酸、ジメチルマロン酸、コハク酸、メチルコハク酸、2,2-ジメチルコハク酸、グルタル酸、アジピン酸、ビメリソ酸との縮合生成物、1-ナフトール、2-ナフトール、ナフタレンジオール、ナフタレントリオール、1-ナフタレンメタノール、2-ナフタレンメタノール、1-(2-ナフチル)エタノール、ナフタレンカルボン酸、1-ナフトール-4-カルボン酸、1,

8 - ナフタレンジカルボン酸、ナフトールスルホン酸などを挙げることができる。また、上記一般式(I I)～(I V)で表わされる置換基は、用いられる露光光の波長に対して吸収の高いものを用いることが好ましい。例えばi線(365 nm)を用いた場合、ベンゾフェノン系置換基が好ましく、K r Fエキシマレーザー(248 nm)を用いた場合、スルホン系あるいはアントラセン系置換基が好ましい。これらの樹脂成分の質量平均分子量は、4000～15000の範囲のものが好ましい。

【0012】また、架橋剤成分としては、加熱により自己同士、あるいは併用する樹脂成分との間で架橋を形成しうる官能基をもつもの、例えばヒドロキシアルキル基又はアルコキシアルキル基あるいはその両方で置換されたアミノ基を少なくとも2個有する含窒素化合物を挙げることができる。このような化合物としては、例えばアミノ基の水素原子がメチロール基又はアルコキシメチル基あるいはその両方で置換されたメラミン、尿素、グアナミン、ベンゾグアナミン、グリコールウリル、スクニルアミド、エチレン尿素などがある。

【0013】これらの含窒素化合物は、例えばメラミン、尿素、グアナミン、ベンゾグアナミン、グリコールウリル、スクニルアミド、エチレン尿素などを沸騰水中においてホルマリンと反応させてメチロール化することにより、あるいはこれにさらにメチルアルコール、エチルアルコール、n-プロピルアルコール、イソプロピルアルコールのような低級アルコールを反応させてアルコキシ化することにより容易に得ることができる。

【0014】これらの含窒素化合物の中で、特に一般式【化5】



(式中のAは水素原子、アルキル基、アラルキル基、アリール基又は $-NR^{11}R^{12}$ 基を示し、 R^7 、 R^8 、 R^9 、 R^{10} 、 R^{11} 及び R^{12} は互いに同じか異なったもので、それぞれ水素原子、メチロール基又はアルコキシメチル基を示すが、分子中に存在する4～6個の R^7 、 R^8 、 R^9 、 R^{10} 、 R^{11} 及び R^{12} の中の少なくとも2個はメチロール基又はアルコキシメチル基である)で表わされる化合物は架橋反応性がよいので好ましい。この一般式で表わされる化合物のメラミン誘導体はメラミン環1個当たり、メチロール基又はアルコキシメチル基を平均3個以上6個未満有するものが好ましい。このようなメラミン誘導体又はベンゾグアナミン誘導体の例としては、市販品のトリアジン環1個当りメトキシメチル基が平均3.7個置換されているMX-750、トリアジン環1個当りメトキシメチル基が平均5.8個置換されているMW

-30(いずれも三和ケミカル社製)や、サイメル300、301、303、350、370、771、325、327、703、712などのメトキシメチル化メラミン、サイメル235、236、238、212、253、254などのメトキシメチル化ブトキシメチル化メラミン、サイメル506、508などのブトキシメチル化メラミン、サイメル1141のようなカルボキシル基含有メトキシメチル化イソブトキシメチル化メラミン、サイメル1123のようなメトキシメチル化エトキシメチル化ベンゾグアナミン、サイメル1123-10のようなメトキシメチル化ブトキシメチル化ベンゾグアナミン、サイメル1128のようなブトキシメチル化ベンゾグアナミン、サイメル1125-80のようなカルボキシル基含有メトキシメチル化エトキシメチル化ベンゾグアナミン(いずれも三井サイアナミッド社製)などが挙げられる。また、グリコールウリルの例としては、サイメル1170のようなブトキシメチル化グリコールウリル、サイメル1172のようなメチロール化グリコールウリルなどが挙げられる。

【0015】また、本発明においては、これらの架橋剤は単独で用いてもよいし、2種以上組み合せて用いてもよい。これらの架橋剤成分は、その質量平均分子量が500～1000のものを用いることが好ましい。

【0016】また、本発明では、上記樹脂成分及び架橋剤成分からなる固形分の質量平均分子量が1300～4500、特に2000～4000であることが好ましい。なお、この範囲を超えるとホール内部にボイドと呼ばれる隙間が生じ、保護膜としての機能を低下させる。また、この範囲を下回るとDenseとIsoの間で膜厚差が生じ、その後のエッチングの制御が非常に難しくなる。この固形分の質量平均分子量は、GPC測定(カラムにはポリスチレン系ゲルを使用し、装置にはSHODEX社製、「GPC SYSTEM-21」)で得られるものである。

【0017】本発明における有機溶剤としては、上記2成分を溶解しうるものであればどのようなものでもよく、特に制限はない。このようなものの例としては、アセトン、メチルエチルケトン、シクロヘキサン、シクロヘキサノン、メチルイソアミルケトン、2-ヘプタノン、1,1,1-トリメチルアセトンなどのケトン類や、エチレングリコール、エチレングリコールモノアセテート、ジエチレングリコール又はジエチレングリコールモノアセテート、あるいはこれらのモノメチルエーテル、モノプロピルエーテル、モノブチルエーテル又はモノフェニルエーテルなどの多価アルコール類及びその誘導体や、ジオキサンのような環状エーテル類や、乳酸エチル、酢酸メチル、酢酸エチル、酢酸ブチル、ビルビン酸メチル、ビルビン酸エチル、3-メトキシプロピオン酸メチル、3-エトキシプロピオン酸エチルなどのエステル類などがある。これらは単独で用いてもよいし、2

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種以上を混合して用いてもよい。

【0018】また、本発明の保護膜形成材料には、塗布性の向上やストリエーション防止のために、所望により界面活性剤を添加することもできる。このような界面活性剤としては、例えばサーフロンSC-103、SR-100（旭硝子社製）、EF-351（東北肥料社製）、フローラードFc-431、フローラードFc-135、フローラードFc-98、フローラードFc-430、フローラードFc-176（住友3M社製）などのフッ素系界面活性剤を挙げることができる。この場合の添加量としては、反射防止膜層の形成用溶液の固形分に対して、2000 ppm未満の範囲で選ぶのが好ましい。

【0019】本発明の保護膜形成材料は、Dense部とIso部からなるパターンを有する基板上に塗布するのに適しているが、このDense部とIso部との関係は、例えば図2に示すように、Dense部が複数個のホールがそのホールの直径の2～3倍程度又はそれ以内の距離に密集し、それから前記の距離以上のある一定の距離を距てて孤立したホールからなるIso部が設けられているものをいう。

【0020】

【実施例】次に実施例、比較例により本発明をさらに詳細に説明する。なお、各例で用いたDense部-Iso部のホールパターンのサンプルは、Dense部として直径250 nmのホール3個が500 nmの間隔で設けられ、Iso部としてDense部の最外部のホールから5000 nm離れて、上記と同じホールを有するものである。

【0021】実施例1

ジヒドロキシフェニルスルホン構造を有するアクリル系樹脂である質量平均分子量が5821のPAC102（ダイトー社製）及びメラミン誘導体である質量平均分子量が599のMX-750（三和ケミカル社製）の6：4の混合物をプロピレングリコールに溶解させ、この固形分濃度を2質量%として保護膜形成材料を調製した。この際の固形分質量平均分子量は4226であった。また、図2に示すようなDense部-Iso部のホールパターンが形成された基板に対して、上記保護膜形成材料をスピナーラー塗布し、180℃にて90秒間ベ*

*一クシ、ホールパターン上に保護膜を形成させた。その断面図をSEM（走査型電子顕微鏡）により観察したところ、パターン内部でのボイドの発生はなく、Dense部-Iso部パターン間での膜厚差は243 nm程度であった。

【0022】実施例2

実施例1で用いたPAC102とMX-750の配合割合を4：6とした以外は、実施例1と同様の操作によりホールパターン上に保護膜を形成させた。この際の固形分質量平均分子量は3235であった。その結果、パターン内にボイドの発生もなく、Dense部-Iso部間の膜厚差は209.5 nm程度であった。

【0023】比較例1

実施例1で用いたPAC102とMX-750の配合割合を8：2とした以外は、実施例1と同様の操作によりホールパターン上に保護膜を形成させた。この際の固形分質量平均分子量は5086であった。その結果、ホール内にボイドが発生したため、完全にホールを埋むことができなかった。

【0024】比較例2

実施例1で用いたPAC102とMX-750の配合割合を1：9とした以外は、実施例1と同様の操作によりホールパターン上に保護膜を形成させた。この際の固形分質量平均分子量は1268であった。その結果、パターン内にボイドの発生はなかったものの、Dense部-Iso部間での膜厚差が425.2 μmとなり、それ以降のリソグラフィー工程の障害となつた。

【0025】

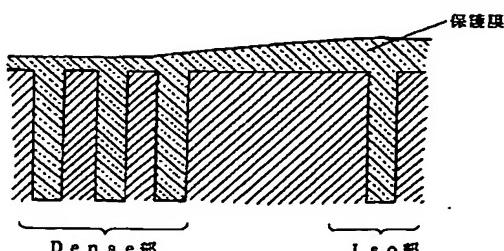
【発明の効果】本発明の保護膜形成材料を用いると、デュアルダマシンプロセスにおいて形成された微細構造レジストパターン上に保護膜を設ける場合に、Dense部とIso部との間に生じる膜厚差を平坦化することができ、後続のトレンチ形成のためのリソグラフィー処理を円滑に行うことができる。

【図面の簡単な説明】

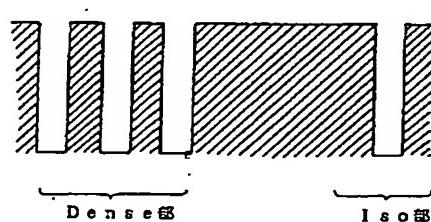
【図1】従来方法における保護膜の状態を示す断面図。

【図2】Dense部とIso部を有するレジストパターンの断面図。

【図1】



【図2】



フロントページの続き

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